

## **APPARATUS AND METHOD FOR DISPENSING HIGH PURITY MATERIALS**

### **Field of the Invention**

**[0001]** This invention relates to system or apparatus and a method for dispensing high purity material from a container without compromising the purity of the material. More particularly, the invention relates to a system or apparatus and a method for dispensing high purity chemicals, and particularly ultra dry solvents intended for use in laboratory scale synthesis studies, from a container, and particularly from a positive dispense container or can while maintaining the integrity of the material.

### **Background to the Invention**

**[0002]** Numerous dispensing systems are known for dispensing materials from pressurized containers. The material to be dispensed in the pressurized container generally provided with a dispensing valve. Upon activation of the valve a pressure differential between the internal pressure in the container and the ambient pressure permits the dispensing of the material from the container. While such a simple system is acceptable for many applications, where the continuing purity and integrity of the material to be dispensed is a prime concern, such systems do not permit the purity and integrity of the material to be maintained upon dispensing of the material. One such material is ultra dry solvent utilized in laboratory scale synthesis studies. Such ultra dry solvents are degraded by contact with moisture and oxygen.

**[0003]** For large scale storage and dispensing of such ultra dry solvents there is presently available a CYCLE-TAINER container for a solvent delivery system. Such system comprises a closed stainless steel container, available in 4, 8, 52, 215 and 1250 liter sizes, containing the material to be dispensed, and is provided with

appropriate valving. In use, an inlet valve of the container is hooked up to a source of pressurized inert gas, such as argon or nitrogen, and the outlet valve from the container is connected to a hose leading directly to the reactor or vessel where the stored material is to be employed or used. Upon introduction of the pressurized gas into the container the material may be dispensed. Cutting off the source of inert gas and closing of the valve stops the dispensing of the material from the container and preserves the integrity of the stored material. Such a system is not suitable, practical or acceptable, however, for conditions where one desires to dispense only small quantities of the material from a positive pressurized container, such as where one desires to dispense from a pressurized container only about 10 to about 20 mL of an ultra dry solvent material for lab scale synthesis studies. One way in which the art has attempted to solve this problem is to employ a syringe and a septum sealed bottle or container that contains, under a blanket of inert gas, the desired material to be dispensed. With this type of apparatus it is necessary for the user to puncture the septum of the bottle with the needle of a syringe to extract the material from the septum container. However, this insertion of the needle into the septum container itself can be a cause of contamination of the material and destroy the integrity of the material. Additionally, the puncturing of the septum leaves a hole in the septum permitting escape of the blanket of inert gas and introduction of atmospheric gas, particularly oxygen, and moisture into the material stored in the septum container. Thus, there is a definite need for an acceptable dispensing system or apparatus for dispensing a material, especially relatively small amount of material, from a pressurized material that permits the dispensing to occur while maintaining the purity and integrity of the material being dispensed.

#### Summary of the Invention

**[0004]** This invention relates to a system or apparatus and a method for dispensing a material from a pressurized container while maintaining product integrity. The system or apparatus comprises a pressurized container for holding

the material and having a dispensing valve provided with a dispensing conduit or stem for dispensing the material from the container at the appropriate time, a syringe equipped with on/off locking mechanism for permitting or preventing flow of the material into and out of the syringe by way of a stem or conduit attached to the locking mechanism, and a removable adaptor for connecting the stem of the syringe to the stem of the dispensing valve of the pressurized container for permitting flow of material from the pressurized container into a chamber of the syringe upon actuation of the dispensing valve.

**[0005]** In a preferred embodiment of the system or apparatus of this invention the dispensing valve comprises a reciprocatably actuated valve wherein, when the syringe stem is connected to the valve stem by way of the adaptor, and the valve stem is reciprocatably moved inward towards the interior of the pressurized container, one or more openings in the valve stem are uncovered permitting flow of material from the container to a chamber of the syringe without exposure to any contaminants or reactive materials thereby permitting dispensing of the material while maintaining its integrity. In another preferred feature of this invention, a dessicant is provided for preventing moisture from entering the pressurized container.

**[0006]** The invention in one embodiment comprises an apparatus for storing and dispensing a material from a pressurized container while maintaining the integrity or purity of the material to be dispensed, the apparatus comprising:

- a) a pressurized container holding the material to be dispensed, the pressurized container having a dispensing valve with a dispensing valve stem having a distal end protruding from the pressurized container and a proximal end extending into the pressurized container;
- b) a syringe provided with 1) an on/off locking member for permitting/or preventing flow of the material into or out of a chamber of the syringe and 2) a stem having an end extending from the syringe for receiving

- material from the pressurized container; and
- c) a removable adaptor connecting the distal end of the dispensing valve stem protruding from the pressurized container to the end of the stem extending from the syringe so as to establish a permissible flow path from one stem to the other stem;
- so that upon activation of the on/off locking member to the on position, for permitting flow of material into the syringe and activation of the dispensing valve to a dispensing mode, material may flow from the pressurized container to the chamber of the syringe.

**[0007]** The invention in another embodiment comprises an apparatus wherein the dispensing valve comprises a reciprocatably actuated valve.

**[0008]** The invention in another embodiment comprises an apparatus wherein the valve stem of the dispensing valve is hollow but is closed at the proximal end in the container and is provided with one or more covered openings along the stem, the valve stem being reciprocatably movable inward into the pressurized container for uncovering the one or more openings in the valve stem for permitting the flow of material from the pressurized container through the one or more openings, into the valve stem, and into the chamber of the syringe.

**[0009]** The invention in another embodiment comprises apparatus wherein the one or more openings in the dispensing valve stem are located circumferentially around the dispensing valve stem and are covered by a gasket until the dispensing valve stem is moved reciprocatably inward into the pressurized container for uncovering the one or more openings.

**[0010]** The invention in another embodiment comprises an apparatus wherein the gasket is a ring of material surrounding the dispensing valve stem in a slidable manner.

**[0011]** The invention in another embodiment comprises an apparatus wherein the pressurized container holding the material to be dispensed is a positive dispense container comprising a pressurized container having therein a bag containing the material to be dispensed, said bag being connected to the dispensing valve in fluid flow relationship, and said bag being surrounded by pressurizing gas, preferably such as argon or nitrogen, in the container.

**[0012]** The invention in another embodiment comprises an apparatus wherein the pressurized container holding the material to be dispensed is a positive dispense container comprising a pressurized container having therein a bag containing the material to be dispensed, said bag being connected to the dispensing valve in fluid flow relationship, and said bag being surrounded by pressurizing gas in the container. In a further embodiment a desiccant material may be placed in the space between the pressurized container and the bag therein holding the material to be dispensed, i.e., in the space where the pressurized gas is maintained between the wall of container and bag to prevent any moisture in the container from entering the bag containing the material to be dispensed.

**[0013]** The invention in another embodiment comprises an apparatus wherein the bag is connected to the dispensing valve through a valve connector, the valve connector having a central opening for permitting the material to flow out of the bag, the dispensing valve stem is seated in the valve connector in a biased manner in a direction outwardly from the pressurized container preventing flow of material out through the valve stem until actuation of the dispensing valve.

**[0014]** The invention in another embodiment comprises an apparatus wherein the dispensing valve stem is seated upon a biasing spring in the valve connector.

**[0015]** The invention in another embodiment comprises an apparatus wherein

the adaptor comprises two female sockets connected by a central material flow path passageway in the adaptor, one of the female sockets connected to the distal end of the dispensing valve stem and the other female socket connected to the end of the stem of the syringe for permitting the flow of the material from the dispensing valve stem to the syringe stem so that the material may flow from the pressurized container to the chamber of the syringe without exposure to the atmosphere.

**[0016]** The invention in another embodiment comprises an apparatus wherein the adaptor comprises two female sockets connected by a material flow path, one of the female sockets connected to the distal end of the dispensing valve stem and the other female socket connected to the end of the stem of the syringe for permitting the flow of the material from the dispensing valve stem to the syringe stem so that the material may flow from the pressurized container to the chamber of the syringe.

**[0017]** The invention in another embodiment comprises an apparatus wherein the on/off locking member on the syringe is a luer lock.

**[0018]** The invention in another embodiment comprises an apparatus wherein the syringe is provided with a reciprocatably actuated piston extending outwardly from the chamber of the syringe at an end of the syringe opposite the syringe stem.

**[0019]** The invention in another embodiment comprises a method for storing and dispensing a material from a pressurized container while maintaining the integrity or purity of the material to be dispensed, the method comprising:

- a) providing a pressurized container holding the material to be dispensed, the pressurized container having a dispensing valve with a dispensing valve stem having a distal end protruding from the pressurized container and a proximal end extending into the pressurized container;
- b) providing a syringe provided with 1) an on/off locking member for permitting/or preventing flow of the material into or out of a chamber of

- the syringe and 2) a stem having an end extending from the syringe for receiving material from the pressurized container; and
- c) providing a removable adaptor for connecting the distal end of the dispensing valve stem protruding from the pressurized container to the end of the stem extending from the syringe so as to establish a permissible flow path from one stem to the other stem;
- d) connecting the adaptor to the distal end of the dispensing valve stem protruding from the pressurized container to the end of the stem extending from the syringe; and
- e) activating the on/off locking member to the on position for permitting flow of material into the syringe;
- f) activating the dispensing valve to a dispensing mode permitting material to flow from the pressurized container to the chamber of the syringe; and
- g) activating the on/off locking member to the off position to isolate the material in the chamber of the syringe.

**[0020]** The invention in another embodiment comprises a method having the additional step of subsequently replacing the stem of the syringe with a needle, activating the on/off locking member to the on position and expelling material from the syringe chamber to a closed system where the material is to be employed.

**[0021]** The invention in another embodiment comprises a method wherein the dispensing valve comprises a reciprocatably actuated valve.

**[0022]** The invention in another embodiment comprises a method wherein the valve stem of the dispensing valve is hollow but is closed at the proximal end in the container and is provided with one or more covered openings along the stem, the valve is actuated by the valve stem being reciprocatably moved inward into the pressurized container thereby uncovering the one or more openings in the valve

stem and permitting the flow of material from the pressurized container through the one or more openings, into the valve stem, and into the chamber of the syringe.

**[0023]** The invention in another embodiment comprises a method wherein the one or more openings in the dispensing valve stem are located circumferentially around the dispensing valve stem and are covered by a slidable gasket and when the dispensing valve stem is moved reciprocatably inward into the pressurized container the one or more openings is/are uncovered by the gasket to provide for flow of the material into the one or more openings.

**[0024]** The invention in another embodiment comprises a method wherein the pressurized container holding the material to be dispensed is a positive dispense container comprising a pressurized container having therein a bag containing the material to be dispensed, said bag being connected to the dispensing valve in fluid flow relationship, and said bag being surrounded by pressurizing gas, preferably argon or nitrogen, in the container, whereby upon activation of the dispensing valve the pressurizing gas forces material from the bag through the dispensing valve and into the syringe chamber.

**[0025]** The invention in another embodiment comprises a method wherein the bag is connected to the dispensing valve through a valve connector, the valve connector having a central opening for permitting the material to flow out of the bag, the dispensing valve stem is seated in the valve connector in a biased manner in a direction outwardly from the pressurized container preventing flow of material out through the valve stem until actuation of the dispensing valve occurs.

**[0026]** The invention in another embodiment comprises a method wherein the dispensing valve stem is seated upon a biasing spring in the valve connector and activation of the dispensing valve causes the biasing spring to be contracted permitting the dispensing valve stem to further enter the pressurized container to



uncover the gasket from the one or more openings circumferentially around the stem.

**[0027]** The invention in another embodiment comprises a method wherein the adaptor comprises two female sockets connected by a central material flow path passageway in the adaptor, and the adaptor is connected to the two stems by connecting one of the female sockets to the distal end of the dispensing valve stem and the other female socket to the end of the stem of the syringe.

**[0028]** The invention in another embodiment comprises a method wherein the material to be dispensed is an ultra dry solvent.

**[0030]** The invention in another embodiment comprises a method having the additional steps of subsequently replacing the syringe stem with a needle while the locking member is in the off position, and then activating the locking member to the on position and expelling material from the syringe chamber to a closed reactor or vessel.

**[0031]** The invention in another embodiment comprises a method wherein the material is expelled from the syringe chamber by means of a reciprocable syringe piston.

#### Brief Description of the Drawings

**[0032]** The invention is illustrated by, but not limited to, the embodiment of the invention shown in the figures wherein:

Fig. 1 is a exploded view, partially in section, of the apparatus or system of this invention;

Fig. 2 is an enlarged view of a portion of the apparatus or system of Fig. 1;

Fig. 3 is a view of the assembled apparatus or system ready for dispensing material from the pressurized container;

Fig. 4 is a view of the assembled apparatus or system of Fig. 3, partially

broken away to show the relative position of the components;

Fig. 5 is an enlarged view of a portion of the assembled apparatus or system of Figs. 3 and 4, illustrating the activation of the valve to permit flow of the material out of the pressurized container; and

Fig. 6 is view illustrating apparatus to illustrate how the material dispensed into the chamber of the syringe by the apparatus of this invention may be dispensed from the syringe.

#### Detailed Description of the Invention

**[0033]** The invention is illustrated by, but not limited to, the following description of preferred embodiments of the invention. As shown particularly in Figs. 1 and 2, the system or apparatus **10** comprises a pressurized container **12** containing the material, particularly an ultra-dry solvent material, to be dispensed. Although any suitable pressurized container can be employed in the apparatus and system of this invention, it is preferred to employ a positive dispense container having a bag of the material in a container pressurized by a suitable pressurizing gas contained in the container but not in the bag. Such positive dispense bags are disclosed, for example in US Patent Nos. 5,035,351 5,040,704 5,137,186 and 5,169,037, the disclosures of which are incorporated herein by reference thereto. As illustrated in Fig. 1, the pressurized container **12** has a material-containing bag **14** in which the material **16** is contained. The bag **14** is exteriorally surrounded by pressurized gas **18** in the container **12**. In an embodiment, any suitable desiccant material **58** may be placed in the space between the wall of the pressurized container **12** and the bag **14**, i.e., in the space occupied by the pressurized gas **18**, to prevent any moisture resident in the container from entering the bag and contaminating material **16**. Any suitable desiccant in any suitable form, e.g., powder or packaged form, may be employed. Preferably the desiccant is a packaged form and comprises packaged silica gel or molecular sieve desiccant, such as those available from Sigma-Aldrich, Inc. of St. Louis, MO, as Minipax® absorbent packets

and from Multisorb Technologies, Inc. of Buffalo, NY as Natrasorb® desiccant packages. The bag **14** containing the material **16** to be dispensed is sealed with a dispensing valve assembly **20** by way of a valve connector **22** suitably sealed to the bag and having a central opening **24** extending through the valve connector. Seated in the central opening **24** of the valve connector **22** is a reciprocable operable hollow valve stem **26**, closed at its inner or bottom proximal end to prevent flow of material directly into the proximal end of the valve stem. That closed end of the valve stem is located in the portion of the valve connector **22** extending into the interior of the container **12** and being seated on a biasing structure **28**, such as a spring, biasing the distal end of the valve stem outward of the container **12** and doing so in a mode preventing dispersing of the material **16** from the bag **14**. The valve stem **26** is provided with one or more circumferentially spaced openings or holes **30** at a location along the valve stem within the central opening **24** of the valve connector **22** located within the interior of the pressurized container **12**. Dispensing of the material **16** from the container **12** through openings or holes **30** is prevented by a covering or gasket **32** surrounding the openings or holes when the valve stem **26** is outwardly biased as shown in Fig. 1 and 2. The gasket material **32** may be of any suitable material not detrimental to the material being stored in the bag **14**. As an example of such a suitable gasket material there may be mentioned Gasket CS05-0310-51 from Buckeye Rubber & Packing Co., Cleveland, Ohio. It will be understood that the gasket material will be chosen based on its suitability to the material **16** to be contained in the bag **14**.

**[0034]** The apparatus or system **10** of the invention further includes an adaptor **34** having a central passageway **36** through the adaptor and thereby providing female sockets **38**, **40** at each end of the adaptor for receiving a male member as explained hereinafter. The adaptor **34** may be of any suitable size or shape suitable for the intended use and made of any material suitably compatible with the material **16** to be dispensed.

**[0035]** The apparatus or system **10** of this invention further comprises a syringe **42** having a central chamber **44** and provided at one end with a reciprocatably actuated piston member **46** extending outwardly from the chamber **44** in a sealing manner and at the other end a on/off mechanism or locking member **48**. Extending outwardly from the locking member **48** is a hollow stem or conduit **50** for receiving material **16** from container **12** into syringe chamber **44** and for dispensing the material from the syringe chamber into a reactor or reaction vessel. The locking member **48** may be a luer lock mechanism or any other suitable on/off mechanism. The locking member **48**, as illustrated in the Figures 1, 3, 4 and 6 has a turnable handle **52**, that by appropriate positioning either prevents or permits flow of material through stem **50** into or out of syringe chamber **44**.

**[0036]** As shown in Figs. 3 and 4, for using the apparatus or system of this invention, the adaptor **34** is connected to both the valve stem **26** of the container **12** and to the stem **50** extending from the syringe **42** by placing the male stems into sealing arrangement with female sockets **38** and **40**, respectively, of the adaptor.

**[0037]** When it is desired to dispense material **16** from bag **14** in container **12**, after the syringe stem **50** and container stem **26** are connected by adaptor **34**, downward pressure is exerted against biasing means **28**, such as by downward movement of the syringe **42**, such that stem **26** is forced further into central opening **24** of valve connector **22** as shown in Fig. 5. Gasket **32** remains stationary against the upper end **23** of valve connector **22** as the stem **26** moves downward compressing biasing spring **28** in the central opening **24** thereby exposing the holes or openings **30** previously covered by the gasket. Upon uncovering of the holes or openings **30**, pressurized gas **18** in container **12** causes material **16** to flow out of bag **14** and through central opening **24** and then through holes or openings **30** into hollow stem **26**, through the adaptor **34**, and then through stem **50** into syringe chamber **44** upon positioning handle **52** of locking member **48** to the on (open) position. Once the material is in the syringe chamber **44** the handle **52** of the locking

member **48** may be turned to the off (closed) position to isolate the material **16** in the chamber.

**[0038]** When it is desired to dispense the material **16** that now resides in the syringe chamber **44** the syringe stem **50** is removed from female socket **38** of the adaptor and the stem is replaced with a syringe needle **54** as illustrated in Fig. 6. The material **16** may then be dispensed from the syringe chamber **44** by placing the needle in a closed reactor or vessel (not shown), turning handle **52** to the on (open) position and expelling the desired amount of the material by appropriate movement of syringe piston **46**.

**[0039]** The invention is particularly applicable to an apparatus and a method for dispensing materials that comprises ultra-dry solvents to be dispensed in relatively small amounts of about 200mL or less, particularly about 100 mL or less, and especially amounts of about 10 ml to about 20 mL, under conditions that require the integrity and impurity of the material to be maintained and exposure to atmospheric conditions or other means of contamination are to be avoided. Examples of such solvent materials include, but are not limited to acetone, acetonitrile, anhydrous alcohols, bis (2-methoxyethyl)ether, chlorobenzene, chloroform, cyclohexane, dimethylformamide, dimethyl sulfoxide, ethyl acetate, ethyl ether, hexanes, methyl tert-butyl ether, methylene chloride, methyl ethyl ketone, tetrahydrofuran, toluene, xylenes and the like

**[0040]** While the invention has been described herein with reference to the specific embodiments thereof, it will be appreciated that changes, modification and variations can be made without departing from the spirit and scope of the inventive concept disclosed herein. Accordingly, it is intended to embrace all such changes, modification and variations that fall within the spirit and scope of the appended claims.